



IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

Kazuhiro Nishikata, et al :GROUP ART UNIT: 1615

SERIAL NO: 09/147,861 :EXAMINER: Amy E Pulliam

FOR: COATED POWDER AND COSMETIC PREPARED BY BLENDING THE SAME

DECLARATION UNDER 37 C.F.R. SECTION 1.132

THE COMMISSIONER OF U.S. PATENT AND TRADEMARK OFFICE

WASHINGTON, D.C. 20231

SIR

Now comes Masaharu Takatori who deposes and states that:

1. I am a graduate of The University of Kyoto, Japan, where I majored in synthetic chemistry.

2. I have been employed by Pola Chemical Industries, Inc. for 27 years, during which period, I was engaged in research of chemical analysis for 10 years, was engaged in development of new materials for 7 years, and thereafter and up to the present I have been working in the Patent Department of said company. At present I am still supervising and giving various advices regarding analysis and development of new materials to persons involved in research and development and guiding them.

3. I have read and understood the Office Action dated April 23, 2003 and the documents cited therein.

4. The following experiments were conducted and performed by me.

COMPARATIVE EXPERIMENTS OF THE COATED POWDER HAVING THE SECOND COATING LAYER FORMED IN AN AMOUNT DIFFERENT FROM THE AMOUNT DEFINED BY THE PRESENT INVENTION AND THE COATED POWDER OF THE PRESENT INVENTION.

[The comparative example 1]

The coated powder described in the following table 1 is made in the same way as the method disclosed in the example 1 of the specification of the present application as follows.

2 wt% aqueous ammonia was gradually added dropwise to a solution of 10 wt% monomethyl-triethoxysilane in ethanol, and the resulting mixture was stirred for 3 hours to produce spherical silica particle (particle size: 0.8  $\mu$ m). This particle was filtered off and washed with water, and then heated and dried at 300°C for 4 hours. The particle was cooled to the room temperature, and then introduced into a solution of 1 wt% titanium isopropoxide in isopropanol. After that, while stirring the resulting mixture under nitrogen atmosphere, 10 ml of isopropanol containing 5% water was gradually added dropwise thereto, thereby depositing titanium hydrolyzate on the surface of the particle. After filtration, the particle was washed with water, and then heated

and dried at 300 °C for 4 hours. This powder was further introduced into a solution of 10 wt% tetraethoxysilane in ethanol, and 1N aqueous hydrochloric acid was gradually added dropwise thereto. The resulting mixture was stirred for one day-and-night to form a silica coating layer on the surface of the particle. The powder finally obtained was burned at 800 °C for 4 hours to obtain a coated powder (A).

By using this coated powder (A), the last part of the above process, "The coated powder was further introduced into a solution of 10 wt% tetraethoxysilane in ethanol, and 1N aqueous hydrochloric acid was gradually added dropwise thereto. The resulting mixture was stirred for one day-and-night to form a silica coating layer on the surface of the particle. The powder obtained was burned at 800 °C for 4 hours" is repeated.

Weight ratio of silica and titanium was measured by a peak intensity ratio originated from titanium dioxide ( $\text{TiO}_2$ ) by X ray diffraction in each coating stage of above-mentioned process.

As a result, when coating of the second coating layer of silica was performed 9 times, the coated powder was obtained, wherein the ratio of silica (powder as a forming core) : titanium (first layer) : silica (second layer) was 23: 2: 75.

Table 1

	silica (core):titanium (first layer):silica (second layer)		
comparative example 1	23	:	2 : 75
comparative example 2	38	:	2 : 60
comparative example 3	48	:	2 : 50

[The comparative example 2]

By using the coated powder (A) obtained in the same way as the comparative example 1, the last part of the above process, "The coated powder was further introduced into a solution of 10 wt% tetraethoxysilane in ethanol, and 1N aqueous hydrochloric acid was gradually added dropwise thereto. The resulting mixture was stirred for one day-and-night to form a silica coating layer on the surface of the particle. The powder finally obtained was burned at 800 °C for 4 hours" is repeated.

When coating of the second coating layer of silica was performed 7 times, the coated powder was obtained, wherein the ratio of silica (powder as a forming core) : titanium (first layer) : silica (second layer) was 38: 2: 60, as shown in Table 1.

[The comparative example 3]

By using the coated powder (A) obtained in the same way as the comparative example 1, the last part of the above process, "The coated powder was further introduced into a solution of 10 wt% tetraethoxysilane in ethanol, and 1N aqueous hydrochloric acid was gradually added dropwise thereto. The resulting mixture was stirred for one day-and-night to form a silica coating layer on the surface of the particle. The powder finally obtained was burned at 800 °C for 4 hours" is repeated.

When coating of the second coating layer of silica was performed 5 times, the coated powder was obtained, wherein the ratio of silica (powder as a forming core) : titanium (first layer) : silica (second

layer) was 48: 2: 50, as shown in Table 1.

[Result of the experiments]

About the coated powder obtained in comparative examples 1 to 3, the experiment of transmission of light was performed in the same way as the method described in the specification of the present application. The result is shown in Table 2 below. Note that, the result given as examples in Table 2 corresponds to the result of examples 1 to 4 described in Table 1 of the specification of the present application.

Table 2

	Linear transmittance (nm)				Total transmittance (nm)			
	400	500	600	700	400	500	600	700
comparative example 1	78	83	86	88	98	99	99	99
comparative example 2	72	76	81	83	99	99	98	99
Comparative example 3	68	73	77	80	98	99	99	99
example 1	15	21	28	36	93	95	94	95
example 2	20	27	34	42	98	99	98	98
example 3	40	50	58	66	99	99	99	99
example 4	53	62	69	73	99	99	99	99

Additionally, the experiment with the foundation contained these coated powder in the same way as the method described in the specification of the present application was also performed.

The foundation that contains each powder at 40 wt % and consists of composition indicated in Table 3 is made. The transmission degree of light of each foundation was measured. The result is shown in Table 4. Note that, the result given as examples in Table 4 corresponds to the result of examples 5 to 8 described in Table 3

of the specification of the present application.

Table 3

Composition	
Coated powder	40 %
Yellow iron oxide	3 %
Red iron oxide	1 %
Methyl paraben	0.2%
Talc	20.8%
Sericite	20 %
Titanium oxide	5 %
Dimethyl polysiloxane	10 %

Table 4

	Linear transmittance (nm)				Total transmittance (nm)			
	400	500	600	700	400	500	600	700
comparative example 1	64	68	72	76	87	88	90	91
comparative example 2	63	66	71	74	83	84	88	90
Comparative Example 3	58	62	64	68	83	88	89	91
Example 5	10	15	19	28	82	85	87	89
Example 6	15	20	22	29	86	89	90	90
Example 7	30	38	41	44	86	90	92	90
Example 8	47	52	63	60	87	91	93	91

All the above-mentioned foundations were used by a woman of age 40's, applied the foundation to her face one after another, and five panelists of women in the ages of 30's-40's observed and evaluated the foundations respectively under the natural light.

#### Evaluation Standards

- X : Received an impression of more unnatural whiteness than the non-made-up face.
- △ : Received an impression of slightly unnatural whiteness than the non-made-up face.
- : Received an impression the same as the non-made-up face.

Table 5

	A	B	C	D	E
comparative example 1	X	X	X	X	X
comparative example 2	X	X	X	X	X
Comparative example 3	△	X	△	△	X
Example 5	○	○	○	○	○
Example 6	○	○	○	○	○
Example 7	○	○	○	○	○
Example 8	○	○	○	○	○

Regarding the luminosity of each of these foundations, no great difference was noted between them.

I hereby declare that all statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of application or any patent issuing thereon.

Date: Oct. 22, 2003

*Masaharu Takatori*  
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